Notebook, N-07-01-28.1

This notebook covers the period January-May 1907. It was used by Edison and laboratory chemist Arthur H. Glaister to record notes regarding the preparation of iron-antimony compounds for storage battery experiments. Several entries note the involvement of Otto Groethe. At the beginning of the book are Edison's instructions to Glaister and what appears to be Glaister's transcription of other Edison instructions. The front cover is labeled "Not very Important." The pages are unnumbered. Approximately 90 pages have been used.

Only the entries listing the instructions for the experiments have been selected.

Glaister
Work on
Antimony
Iron
compounds.

William H. Hunt
... STATIONER ...
Good Work Promptly
68 William Street,
New York.

Standard figures

80% Fe 20% Sb

1781 capacity down to 1 volt.

76.9% ampere efficiency

54.4 watt efficiency

Regular mix

1415 capacity - 1 volt

76.6 ampere efficiency

55.8 watt efficiency

Jany 28 1907

Glansler

I want you to work on Iron Antimony Combinations —

Copy from Dr Grothes book what I say about Iron Salts & Oxides etc

Make 4 oz samples of dry mix Containing always 80% Iron and 20% Antimony —

This 4 oz of dry mix goes to Silver Lake to be reduced by Hydrogen —

½ of the samples will be reduced & self heated the other ½ will be reduced to the non pyrophoric state,

Two pockets of Each half will be made & tested and

the results given to you
so you can see the results,

Put down in this book clearly
+ plainly just how each
sample is made so that it
can without doubt be
exactly duplicated should
the results be good —

Hundreds of combinations can
be made by use of different
oils of Thor + Antimony —

Try every kind of Iron &
Antimony salt & combine them
in various ways.
By precipitating together by igniting
the oxalates together with the Antimony
salts
By moistening ignited Fe_2O_3 with
the salt of Antimony.
By fusing sulphate of Iron with
Potassium sulphate until the SO_3
practically disappears & exhausting
the soluble matter
This oxide is the purest finest
we so far have obtained & is
better than the Iron from oxalates
The Fe_2O_3 sulphate oxide can
then be moistened with the
~~Tartar emetic~~ Antimony salt
There may be some virtue in the
oxide from fusing Ferrous Potassium
sulphate
Also fusing other iron salts with
nitrates chlorides etc to get an Iron
oxide in different physical states
Also double salts of Iron Antimony
Iron Sodium & Antimony Iron Potassium
Antimony fused & exhausted

Commercially we can no longer use oxalates as oxalic acid is too high, we shall use the oxide from fusion of Ferrous Sodium Sulphate which is a cheap process.

You in your experiments bear in mind that the value of the final result will depend upon how cheap it can be made. Our iron pockets so far have an economy running from 40% Efficiency up to 71%
And 440 Milliampere hours per gram in 2 of mixture including the 5% of H_2O

You need make only 40 of dry materials ready to go into the hydrogen tube 30% Fe 20% Ti.
½ of the sample should be reduced in H_2 only heated 5% H_2O added
The other ½ is to be reduced to the non hydrophyric state & 5% H_2O added
2 pockets made for each sample — 5 grammes in each
I will have Thorpe & Whitehead send your results of tests to that

you can see if you are on the
right track.
Any chemicals you want
order through Mr Randolph.

Preliminary experiments

① Saw in Watts that Pb_2O_3 was soluble in a strong slightly acid solution of Fe_2Cl_6 and whether the same applied to the metal.
Made a strong solution of Fe_2Cl_6 & added a trace of HCl then digested with some powdered metallic Pb the metal was attacked readily & went into solution as $PbCl_2$ at the same time reducing the Fe_2Cl_6 to $FeCl_2$ this makes a nice way to get the metal into solution along with Fe or adding H_2O the PbOCl is thrown down in abundance.

② Took some of the Fe_2Cl_6 solution of Pb which was fairly saturated consequently mostly Fe^{II} poured into an excess of Na_2CO_3 solution thus precipitating $FeCO_3 + Pb_2(CO)_3$ warmed the beaker & introduced a stream of air to oxidise the Fe formed

the reaction to be too slow to be of use commercially

(3) Precipitated some more of the solution with Na_2CO_3 & started to filter as it was fairly good to filter but the pH continued considerable fire

(4) Gradually introduced the alloy Yb into a crucible of fused KNO_3 such the mixture of making $KYbO_3$ the Yb was attacked with violence cooled & dissolved out with water, found that there was a lot of Yb in solution as $KYbO_3$ but at the same time there was a lot precipitated probably as Yb_2O_3 or Yb_2O_5 added strong Na_2S solution to the clear portion got an abundant ppt of Na_3YbO_3 showing that the Yb was present in the form of a meta antimonate

(5) Mixed some of the $KYbO_3$ solution with an excess of a slightly acid solution of Fe_2Cl_6 then poured into an excess of Na_2CO_3

and brought to the boil filtered added HCl to the filtrate and passed H_2S no ppt showing that all the Pb had been carried down by the Fe as Ferric Antimonate $Fe_2(SbO_3)_6$

(5) Made another fusion of metallic Sb this time used a mixture of KNO_3 & K_2CO_3 there was still a good deal insoluble in water though less than with the KNO_3 alone

4 Dissolved some metallic Sb in strong HNO_3 washed the Sb_2O_5 by decantation & digested with a large excess of 33% KOH the precipitate in time all dissolved except a trace which looked like mostly Fe. this solution when mixed with HCl gave much more ppt than the previous solutions

(8) Found Sb bl. with enough HCl present to keep clear solution does not ppt with $K_4Fe(CN)_6$

Results obtained with Na_2CO_3
Ferrous sulphate + Antimony + Lead Iodide
Glauber No 1

Sp. Gr. Acid 5 grams
½ vol Iodine 1 vol ~~Iodine~~
885 1255 1280 865 1220 1045
1160 1500 1635 1100 1095 1140

Non Oxygen Force 5.5 grams
5.5 grams
½ vol ~~Iodine~~ 1 vol Iodine
875 1460 1850 860 960 1120
800 1620 2070 745 1080 1215

In efficiency

Preparation Glauber No 1

Took 792 grams of $FeSO_4$ crystals & 40 grams Sb metal this represents a proportion of 80 – 20
Dissolved the $FeSO_4$ in water ~~& oxydised with HNO_3 specg~~ cc added 70 cc conc. H_2SO_4 then heated to almost boiling & oxidised with HNO_3 it took about 200 cc then added 500 cc in excess making 250 cc in all then added the 40 grams of Sb in fine powder & kept hot on the steam bath with constant stirring it was very slow to dissolving added another 50 cc of ~~Sb~~ / ~~to~~ HNO_3 & another 20 of H_2SO_4 evaporated down to dryness & took up again with water there was still a little Sb left undissolved but let that go poured in an excess of a ~~hot~~ solution of Na_2CO_3 and washed by decantation 10 times filling the basin up